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5-Bromo-1-(4-fluorophenyl)-1,3-dihydroisobenzofuran

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.079
Data-to-parameter ratio = 17.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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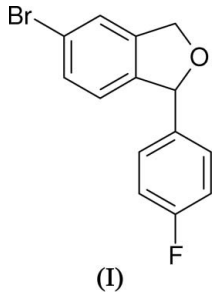
The title compound, $C_{14}H_{10}BrFO$, possesses normal geometrical parameters. The dihedral angle between the two ring systems is 71.50 (9)°. An unusually short intermolecular Br···Br contact of 3.4311 (5) Å occurs.

Comment

The title compound, (I), is an intermediate in the synthesis of the antidepressant drug citalopram (Liechti *et al.*, 2000). More generally, phthalans show distinctive redox chemistry (Azzena *et al.*, 1996). We have previously deposited (CSD-260624; Cambridge Structural Database; Allen, 2002) data for a poor quality structure from a twinned crystal of (I).



(I)

The geometrical parameters for (I) are normal. Each molecule of (I) is chiral (the arbitrarily chosen asymmetric unit has an *S* conformation at C7), but crystal symmetry generates a racemic mixture of the two enantiomers. The nine-membered isobenzofuran ring system (C7–C14/O1) is almost planar [r.m.s. deviation from the mean plane = 0.018 Å; maximum = 0.038 (3) Å for C14] and the dihedral angle between the two ring systems (C7–C14/O1 and C1–C6) is 71.50 (9)°.

A *PLATON* (Spek, 2003) analysis of (I) identified two possible C—H···F interactions (Table 1) that may help to stabilize the crystal packing (Fig. 2). There are no significant π–π stacking interactions in (I).

Inversion symmetry generates a short intermolecular Br1···Br1^{i} [symmetry code: (i) $2-x, -y, 1-z$] separation of 3.4311 (5) Å which is significantly less than the van der Waals contact distance of 3.70 Å for two Br atoms (Bondi, 1964). Some workers have ascribed specific attractive forces to such short intermolecular halogen–halogen contacts (Desiraju & Parthasarathy, 1989). A database survey of such contacts by Eriksson & Hu (2001) shows that the present separation lies at the lower end of the observed range of intermolecular Br···Br distances. However, these workers are less certain of the nature of such contacts, and suggest that they may be the consequence – rather than the cause – of the crystal packing.

In the related 1-(4-fluorophenyl)-1,3-dihydroisobenzofuran-5-carbonitrile [*i.e.* where a cyanide group replaces the Br atom in (I)], there are two molecules in the asymmetric unit with distinctly different degrees of twist between their ring systems (Yathirajan *et al.*, 2004).

Experimental

5-Bromo-3*H*-isobenzofuran-1-one (2.13 g, 10 mmol) was subjected to a Grignard reaction with 4-fluorophenyl magnesium bromide (2.4 g, 12 mmol) in tetrahydrofuran (10 ml) at 273 K. The resulting product was treated with sodium borohydride (0.37 g, 10 mmol) in methanol (10 ml) to obtain the diol, which was cyclized with *p*-toluene sulfonic acid (1 g, 5.81 mmol) in toluene (10 ml) at 353 K, yielding crude (I). Diffraction-quality crystals were obtained by recrystallization from *n*-hexane (Bigler *et al.*, 1977) (m.p. 318 K).

Crystal data

$C_{14}H_{10}BrFO$
M_r = 293.13
Monoclinic, $P2_1/c$
a = 6.0560 (3) Å
b = 7.8659 (4) Å
c = 24.2289 (14) Å
β = 92.542 (3)°
V = 1153.03 (11) Å^3
Z = 4
D_x = 1.689 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2707 reflections
θ = 1.0–27.5°
μ = 3.56 mm^{-1}
T = 120 (2) K
Block, yellow
0.24 × 0.15 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.482, T_{max} = 0.718
12389 measured reflections
2630 independent reflections
1742 reflections with $I > 2\sigma(I)$
R_{int} = 0.072
θ_{max} = 27.6°
$h = -7 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = -31 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.079
S = 1.02
2630 reflections
155 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0257P)^2 + 0.4294P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0023 (6)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9···F1^{i}	0.95	2.54	3.324 (4)	140
C14—H14B···F1^{ii}	0.99	2.52	3.265 (4)	132

Symmetry codes: (i) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$.

The H atoms were positioned geometrically, with C—H = 0.95–0.99 Å, and refined as riding, with U_{iso}(H) = 1.2U_{eq}(carrier).

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997), and *SORTAV* (Blessing, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

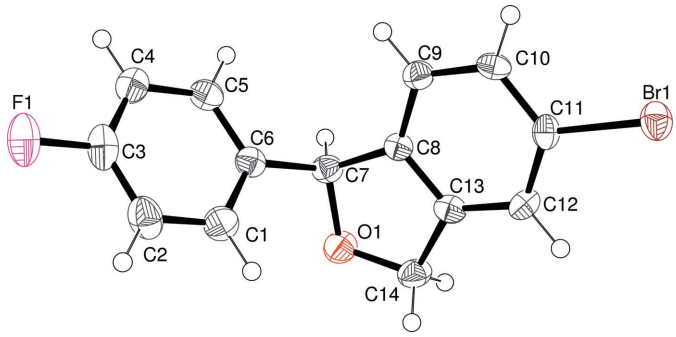


Figure 1
View of (I), showing 50% displacement ellipsoids and arbitrary spheres for the H atoms.

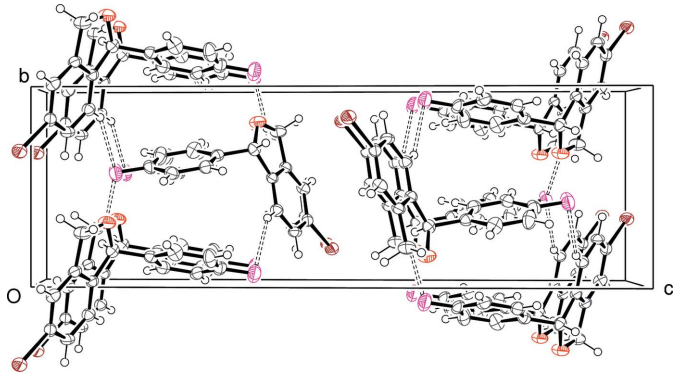


Figure 2
Unit-cell packing in (I), viewed down [100], showing 50% displacement ellipsoids and arbitrary spheres for the H atoms, with short C—H···F interactions shown as dashed lines.

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supporting information

Acta Cryst. (2006). E**62**, o1534–o1535 [https://doi.org/10.1107/S1600536806009810]

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$Z = 4$
$F(000) = 584$
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Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2707 reflections
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$h = -7 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = -31 \rightarrow 27$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.079$
$S = 1.02$
2630 reflections
155 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0257P)^2 + 0.4294P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.56$ e Å^{-3}
$\Delta\rho_{min} = -0.40$ e Å^{-3}
Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0023 (6)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3615 (5)	0.6855 (5)	0.26210 (15)	0.0366 (9)
H1	0.5049	0.7253	0.2731	0.044*
C2	0.3091 (6)	0.6559 (5)	0.20662 (15)	0.0420 (9)
H2	0.4144	0.6748	0.1794	0.050*
C3	0.1012 (6)	0.5986 (4)	0.19235 (14)	0.0357 (9)
C4	−0.0572 (6)	0.5688 (4)	0.22975 (14)	0.0335 (8)
H4	−0.2002	0.5291	0.2183	0.040*
C5	−0.0007 (5)	0.5990 (4)	0.28501 (14)	0.0311 (8)
H5	−0.1070	0.5790	0.3119	0.037*
C6	0.2072 (5)	0.6577 (4)	0.30177 (13)	0.0274 (7)
C7	0.2636 (5)	0.6902 (4)	0.36229 (13)	0.0276 (7)
H7	0.1256	0.7199	0.3813	0.033*
C8	0.3774 (5)	0.5441 (4)	0.39220 (12)	0.0232 (7)
C9	0.3061 (5)	0.3792 (4)	0.39959 (13)	0.0274 (8)
H9	0.1672	0.3430	0.3841	0.033*
C10	0.4407 (5)	0.2669 (4)	0.43009 (13)	0.0263 (8)
H10	0.3949	0.1529	0.4356	0.032*
C11	0.6416 (5)	0.3234 (4)	0.45219 (12)	0.0258 (7)
C12	0.7142 (5)	0.4893 (4)	0.44546 (13)	0.0272 (8)
H12	0.8528	0.5262	0.4610	0.033*
C13	0.5774 (5)	0.5985 (4)	0.41530 (13)	0.0260 (7)
C14	0.6066 (5)	0.7837 (4)	0.40280 (15)	0.0334 (8)
H14A	0.6119	0.8516	0.4373	0.040*
H14B	0.7449	0.8029	0.3834	0.040*
O1	0.4186 (4)	0.8294 (3)	0.36828 (10)	0.0370 (6)
F1	0.0466 (3)	0.5716 (3)	0.13736 (8)	0.0472 (6)
Br1	0.82479 (5)	0.17116 (4)	0.494648 (14)	0.03340 (14)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0242 (17)	0.046 (2)	0.040 (2)	0.0044 (16)	0.0043 (15)	0.0050 (18)
C2	0.040 (2)	0.056 (3)	0.031 (2)	0.0112 (19)	0.0092 (16)	0.0047 (19)
C3	0.049 (2)	0.030 (2)	0.028 (2)	0.0183 (17)	−0.0033 (18)	−0.0054 (16)
C4	0.037 (2)	0.026 (2)	0.036 (2)	0.0035 (15)	−0.0035 (17)	−0.0044 (16)
C5	0.0331 (19)	0.0271 (19)	0.034 (2)	0.0003 (15)	0.0055 (16)	0.0012 (15)

C6	0.0274 (17)	0.0232 (18)	0.0316 (19)	0.0038 (14)	0.0022 (14)	0.0021 (15)
C7	0.0250 (17)	0.031 (2)	0.0277 (19)	−0.0014 (15)	0.0053 (14)	−0.0019 (15)
C8	0.0251 (17)	0.0210 (18)	0.0237 (18)	0.0002 (14)	0.0026 (13)	−0.0020 (14)
C9	0.0272 (17)	0.027 (2)	0.0277 (19)	−0.0025 (14)	0.0015 (14)	−0.0014 (14)
C10	0.0296 (18)	0.0182 (17)	0.032 (2)	−0.0031 (14)	0.0058 (15)	−0.0008 (14)
C11	0.0278 (16)	0.0277 (18)	0.0220 (17)	0.0078 (15)	−0.0002 (13)	−0.0012 (15)
C12	0.0214 (16)	0.028 (2)	0.032 (2)	0.0000 (14)	−0.0025 (14)	−0.0051 (15)
C13	0.0306 (18)	0.0206 (18)	0.0270 (19)	−0.0013 (14)	0.0054 (15)	−0.0037 (14)
C14	0.0291 (19)	0.025 (2)	0.046 (2)	−0.0034 (14)	−0.0020 (17)	0.0010 (16)
O1	0.0387 (13)	0.0231 (13)	0.0484 (16)	−0.0017 (11)	−0.0072 (11)	0.0028 (11)
F1	0.0592 (14)	0.0534 (14)	0.0284 (12)	0.0208 (10)	−0.0033 (10)	−0.0057 (10)
Br1	0.0392 (2)	0.0276 (2)	0.0330 (2)	0.00621 (16)	−0.00337 (14)	0.00021 (16)

Geometric parameters (Å, º)

C1—C2	1.387 (5)	C8—C13	1.380 (4)
C1—C6	1.387 (4)	C8—C9	1.381 (4)
C1—H1	0.9500	C9—C10	1.392 (4)
C2—C3	1.367 (5)	C9—H9	0.9500
C2—H2	0.9500	C10—C11	1.381 (4)
C3—C4	1.369 (5)	C10—H10	0.9500
C3—F1	1.375 (4)	C11—C12	1.389 (4)
C4—C5	1.388 (4)	C11—Br1	1.903 (3)
C4—H4	0.9500	C12—C13	1.380 (4)
C5—C6	1.385 (4)	C12—H12	0.9500
C5—H5	0.9500	C13—C14	1.500 (4)
C6—C7	1.513 (4)	C14—O1	1.428 (4)
C7—O1	1.445 (3)	C14—H14A	0.9900
C7—C8	1.509 (4)	C14—H14B	0.9900
C7—H7	1.0000		
C2—C1—C6	120.8 (3)	C13—C8—C7	109.4 (3)
C2—C1—H1	119.6	C9—C8—C7	129.7 (3)
C6—C1—H1	119.6	C8—C9—C10	119.1 (3)
C3—C2—C1	118.0 (3)	C8—C9—H9	120.5
C3—C2—H2	121.0	C10—C9—H9	120.5
C1—C2—H2	121.0	C11—C10—C9	119.1 (3)
C2—C3—C4	123.6 (3)	C11—C10—H10	120.4
C2—C3—F1	118.3 (3)	C9—C10—H10	120.4
C4—C3—F1	118.0 (3)	C10—C11—C12	122.3 (3)
C3—C4—C5	117.3 (3)	C10—C11—Br1	119.3 (2)
C3—C4—H4	121.3	C12—C11—Br1	118.3 (2)
C5—C4—H4	121.3	C13—C12—C11	117.5 (3)
C6—C5—C4	121.5 (3)	C13—C12—H12	121.3
C6—C5—H5	119.3	C11—C12—H12	121.3
C4—C5—H5	119.3	C8—C13—C12	121.1 (3)
C5—C6—C1	118.7 (3)	C8—C13—C14	109.2 (3)
C5—C6—C7	120.3 (3)	C12—C13—C14	129.7 (3)

C1—C6—C7	121.0 (3)	O1—C14—C13	105.3 (2)
O1—C7—C8	104.4 (2)	O1—C14—H14A	110.7
O1—C7—C6	110.1 (2)	C13—C14—H14A	110.7
C8—C7—C6	114.5 (2)	O1—C14—H14B	110.7
O1—C7—H7	109.2	C13—C14—H14B	110.7
C8—C7—H7	109.2	H14A—C14—H14B	108.8
C6—C7—H7	109.2	C14—O1—C7	111.5 (2)
C13—C8—C9	120.8 (3)		

C6—C1—C2—C3	0.0 (5)	C13—C8—C9—C10	−1.0 (5)
C1—C2—C3—C4	−0.1 (5)	C7—C8—C9—C10	−178.7 (3)
C1—C2—C3—F1	178.9 (3)	C8—C9—C10—C11	0.1 (4)
C2—C3—C4—C5	−0.1 (5)	C9—C10—C11—C12	0.5 (5)
F1—C3—C4—C5	−179.1 (3)	C9—C10—C11—Br1	179.3 (2)
C3—C4—C5—C6	0.3 (5)	C10—C11—C12—C13	−0.2 (5)
C4—C5—C6—C1	−0.4 (5)	Br1—C11—C12—C13	−179.0 (2)
C4—C5—C6—C7	179.7 (3)	C9—C8—C13—C12	1.3 (5)
C2—C1—C6—C5	0.2 (5)	C7—C8—C13—C12	179.5 (3)
C2—C1—C6—C7	−179.9 (3)	C9—C8—C13—C14	−177.4 (3)
C5—C6—C7—O1	−147.9 (3)	C7—C8—C13—C14	0.8 (3)
C1—C6—C7—O1	32.2 (4)	C11—C12—C13—C8	−0.8 (4)
C5—C6—C7—C8	94.9 (3)	C11—C12—C13—C14	177.7 (3)
C1—C6—C7—C8	−85.0 (4)	C8—C13—C14—O1	−3.6 (3)
O1—C7—C8—C13	2.3 (3)	C12—C13—C14—O1	177.8 (3)
C6—C7—C8—C13	122.7 (3)	C13—C14—O1—C7	5.2 (3)
O1—C7—C8—C9	−179.7 (3)	C8—C7—O1—C14	−4.7 (3)
C6—C7—C8—C9	−59.3 (4)	C6—C7—O1—C14	−128.1 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9···F1^{i}	0.95	2.54	3.324 (4)	140
C14—H14*B*···F1^{ii}	0.99	2.52	3.265 (4)	132

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